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Metrology of MID offset mirrors before and after coating

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ABSTRACT

The Materials Imaging and Dynamics (MID) station is located at the SASE2 undulator beamline of European XFEL and has become operational in 2019. The MID instrument operates in the medium to hard X-ray range (5 - 25 keV) and its scientific focus is on time-resolved coherent X-ray scattering and diffraction studies in materials science, with particular interest in ultrafast pump-probe experiments where the pump can be either X-rays, an optical laser beam or a pulsed magnetic field. The optical setup of the MID instrument includes two vertically offset mirrors equipped with cryogenic cooling. The top mirror will be employed for grazing incidence experiments on liquid surfaces, and the bottom mirror will be used to spatially overlap two split beams generated by a "split and delay" line. The mirrors are 500 mm long and are coated with boron carbide (B_4C) and platinum (Pt) in two adjacent stripes. Deterministic mirror polishing is done to compensate the gravitational sag in order to achieve a perfectly flat mirror when it is installed. The requirements were very challenging for the surface shape and the needed ion-beam deterministic polishing, so also the coating process had to be performed and monitored with particular care. We present the metrological characterization of the mirrors, carried out before and after the B_4C and Pt coating, and performed with a large aperture Fizeau interferometer. The measurements were made at European XFEL's metrology lab in grazing incidence setup and with the mirrors facing sideways. Analyzing these data, we can deduce many important parameters, as the peak-to-valley of the central profile, the bending radius, and the gravity compensation profile. We show metrological results before and after coating and give details about the calculations performed to decide whether the mirror shapes are still complying with specifications after all these processes.

Keywords: metrology, coating, Fizeau interferometer, MID instrument, X-rays

1. INTRODUCTION

The European X-ray Free Electron (European XFEL) is a 3.4 km long research facility in the area of Hamburg, built underground. Exceptional capabilities of this facility are the high average brilliance ($1.6\cdot10^{25}$ photons / s / mm^2 / mrad^2/ 0.1% bandwidth), short pulse (in the femtoseconds scale) and extraordinary repetition rate of 27000 flashes per second that make its design unique among the X-ray sources currently in operation all over the world (Ref. 1). The facility started operation in September 2017 with reduced performance but it is approaching quickly the design parameters. In order to get such a performance, the required level of accuracy and reproducibility for the reflective X-ray optics is very high, starting from beam transport mirrors in the tunnels and ending with focusing systems in the experiments hutches. For the flat mirrors in the beam transport, the most common specifications are a surface quality error better than 2 nm P-V and a radius of curvature longer than 5640 km, corresponding to a maximum value for the sagittal of 20 nm on the best fitted (Ref. 2). Similar quality level is required even in the focusing mirrors, like K-B systems installed at the experimental hutches in soft and hard X-ray beamlines in the experiments hutches.

The quality of the surface is not the only important parameter in the manufacturing of European XFEL mirrors. Considering the high power of the beam, there is a high chance to induce a surface damage if the beam is accidentally focused on it. Even if not focused, the power density could be very high and could produce some damage in single or multi shot regime. All the mirrors in European XFEL are cooled, but this is preventing only thermal drift or very long-time heating of the mirror, with consequent drifts of the beam pointing and distortion of the wavefront phase, while the single shot and multi shot damage process is on a much shorter time scale. The duration of the pulses is so short that the damage threshold is defined only by the energy power density, the material of the coating and the photon energy.

Another important parameter for the choice of the material is the reflectivity for a certain range of energies of X-rays. With high energies, the low-Z materials that are used to protect the surface are not reflecting anymore or they

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require a too small grazing incidence angle. Metallic coatings have a better overall reflection with higher incidence angles, and the absorbed energy is distributed to a thicker material so the overall effect is advantageous. This is mandatory when higher photon beam energies are foreseen, more than 20 keV for example. In European XFEL we have usually Boron-Carbide (B_4C) as protective coating, and Platinum (Pt) or Ruthenium (Rh) for high photon energies. The two stripes are displaced along the mirror, in this way it is possible to use either one or the other with a rigid displacement.

There are not only focusing or flat mirrors in European XFEL, but also various other mirrors used in the experimental hutches. The optical setup of the Materials Imaging and Dynamics (MID) instrument includes two vertically offset mirrors that will be employed for grazing incidence experiments on liquid surfaces and to spatially overlap two split beams generated by a “split and delay” line. The mirrors are 500 mm long and are coated with B_4C and Pt in two adjacent stripes. Deterministic mirror polishing is done to compensate the gravitational sag in order to achieve a perfectly flat mirror when it is installed.

The requirements were very challenging for the surface shape and the ion-beam deterministic polishing, so also the coating process had to be performed and monitored with particular care. We want to show here some remarkable results about the surface finishing of these mirrors, measured before and after the coating. Such careful check is really necessary in case of European XFEL optics, because of the high quality of the polished surfaces that could be lost completely after the coating procedure.

2. MIRRORS SPECIFICATIONS AND MEASUREMENTS SETUP

The mirrors specifications are listed in Tab. 1.

Table 1. Specifications table for the elliptical mirrors, in horizontal and vertical orientation.

Specifications		
Mirror ID:	**15-0137 M1**	**15-0137 M2**
Physical dimensions (Length x Width x Thickness)	500 mm x 40 mm x 50 mm	500 mm x 40 mm x 50 mm
Shape	Flat Deterministic polishing to correct gravitational sag	Flat Deterministic polishing to correct gravitational sag
Optical area (Length x Width)	440 mm x 20 mm	440 mm x 20 mm
Tangential shape error	< 2 nm P-V	< 2 nm P-V
Material	Single crystal silicon 110	Single crystal silicon 110
Orientation in operation	Facing up	Facing down

The mirrors are polished by Carl ZEISS SMT GmbH, while the project of the mechanical mounting, motorized support and split and delay line where the mirrors are installed is provided by AXILON GmbH. The specifications are required to imprint a gravitational sag correction on the surface shape in order to have a perfect flat surface when the mirror is installed. The calculated Finite Element Analysis (FEA) are reported in Fig. 1 and Fig. 2 and they are calculated using the mechanical model of the mirror mount.

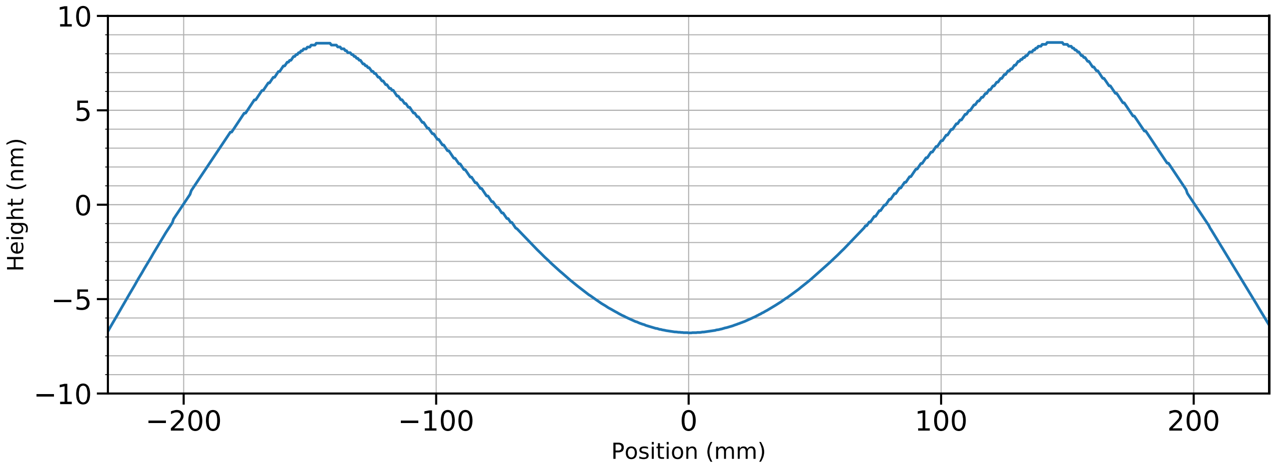

Figure 1. Finite Element Analysis for the 15-0137 M1 mirror, facing up. This is the expected deformation when applied to a flat mirror, and it is the base for calculation of an opposite polishing on the mirror surface.

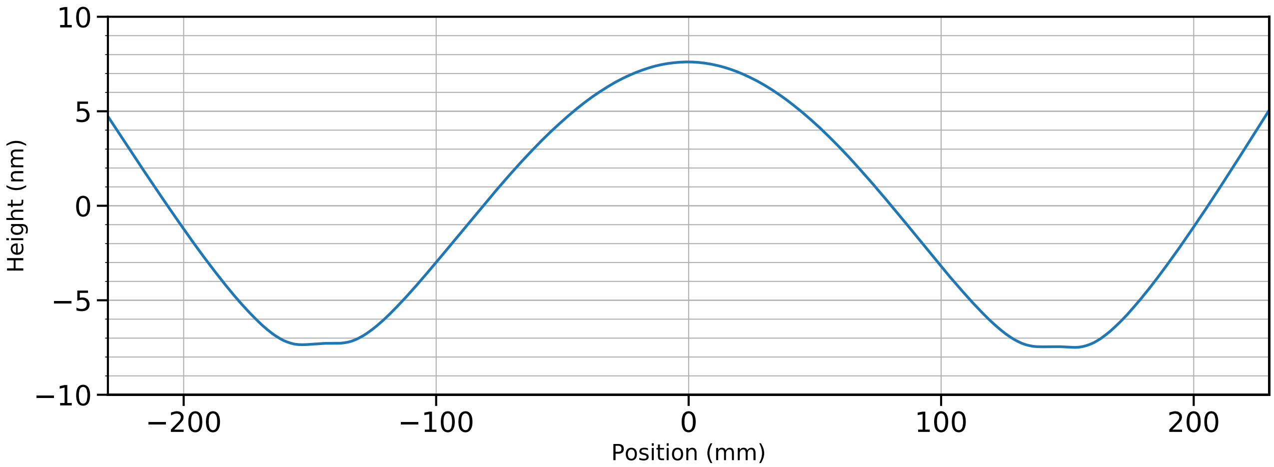

Figure 2. Finite Element Analysis for the 15-0137 M2 mirror, facing down. This is the expected deformation when applied to a flat mirror, and it is the base for calculation of an opposite polishing on the mirror surface. For clarity, the coordinate system is similar to the Fig. 3, with the mirror surface facing upwards.

To have the mirrors on a flat shape when installed, a reciprocal correction compared to the corresponding FEA is imprinted on the surface using ion-beam figuring. The mirrors are measured in side-facing setup, so this is the shape that is measured in reality. It is very impractical to measure the mirrors in the final mount, that's why it is preferable to measure in this way and compare the result with FEA calculations rather than measuring in the final setup.

3. MEASUREMENTS BEFORE COATING

The optical metrology of the surfaces is performed using a Large Fizeau Interferometer. During measurement, the mirror is lying facing side, inserted inside an angled optical cavity and measured interferometrically. The transmission flat (TF) is attached on the Fizeau while the reference flat (RF) is placed on a mechanical mount. The mirror is sitting on three points and imaged in full size on the camera of the interferometer where it is measured (Figure 3). Phase shift measurements are taken in a row of 10, with 250 measurements averaging. Calibration maps of TF and RF are applied. A digital mask corresponding to clear aperture is considered and the central profile is analysed.

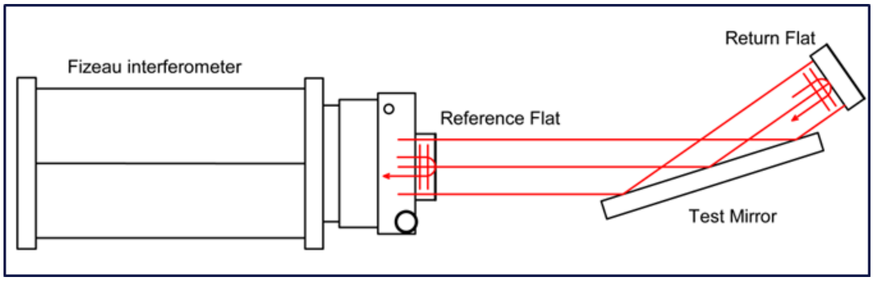

Figure 3. Scheme of the measurement set-up.

A good level of the reference surfaces TF and RF is previously achieved using the three-flat test. The complete measurement is taking some hours to become less sensitive to air fluctuations. An example of measurement is in Fig. 4.

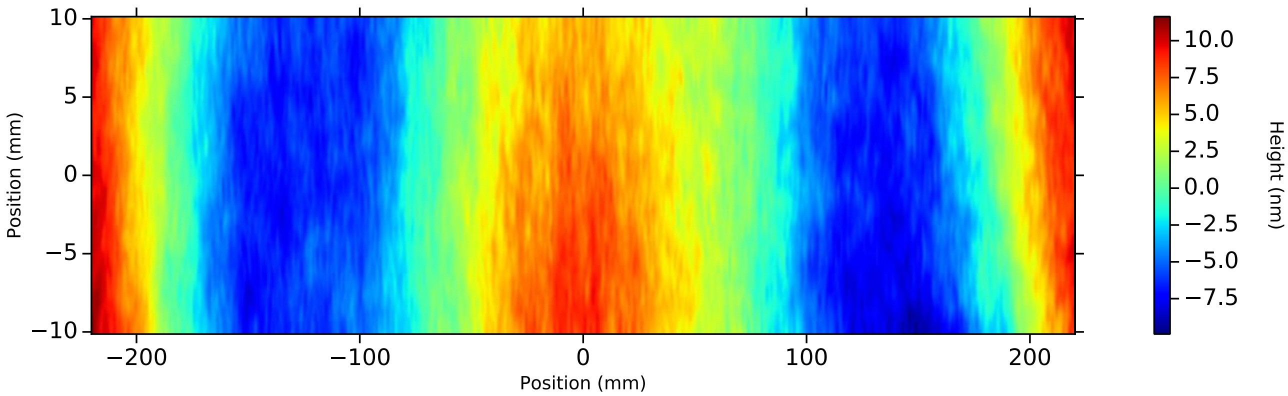

Figure 4. Measurement of the 15-0137 M1 mirror as 2D map, facing side. We can see the imprint on the surface to counterbalance the expected gravity sag when placed facing up.

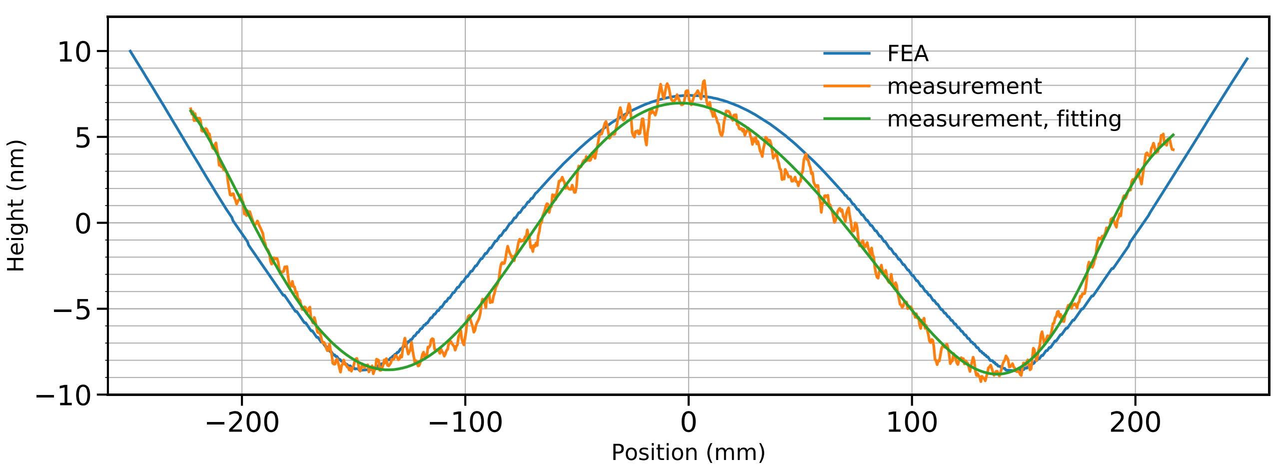

Figure 5. Measurement of the 15-0137 M1 mirror as central profile. For comparison, a polynomial fitting of the profile and the inverse of FEA is also reported. The measurement is clipped on the real clear aperture that is a bit smaller than the calculated FEA.

From the 2D map, a central profile is retrieved and compared with the corresponding FEA (Fig. 5). We adjusted the second order polynomial contribution to let the measurement and the FEA match as much as possible, only for comparison purposes. When we remove the linear fitting from the measurement, we remain with the high frequency profile (Fig. 6 and Fig. 7).

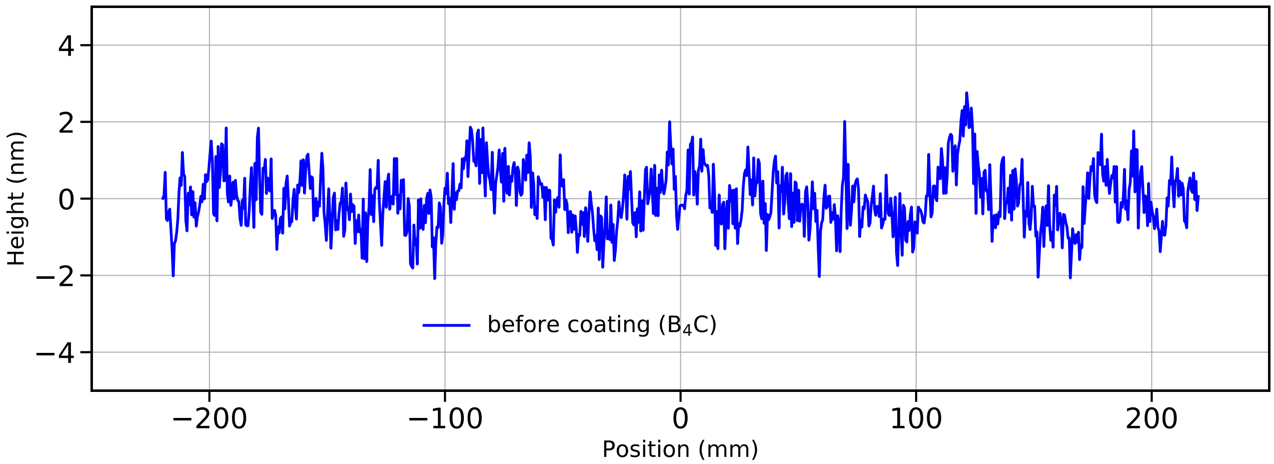

Figure 6. Central profile of the 15-0137 M1 surface before the coating, when 6-power polynomial fitting is removed.

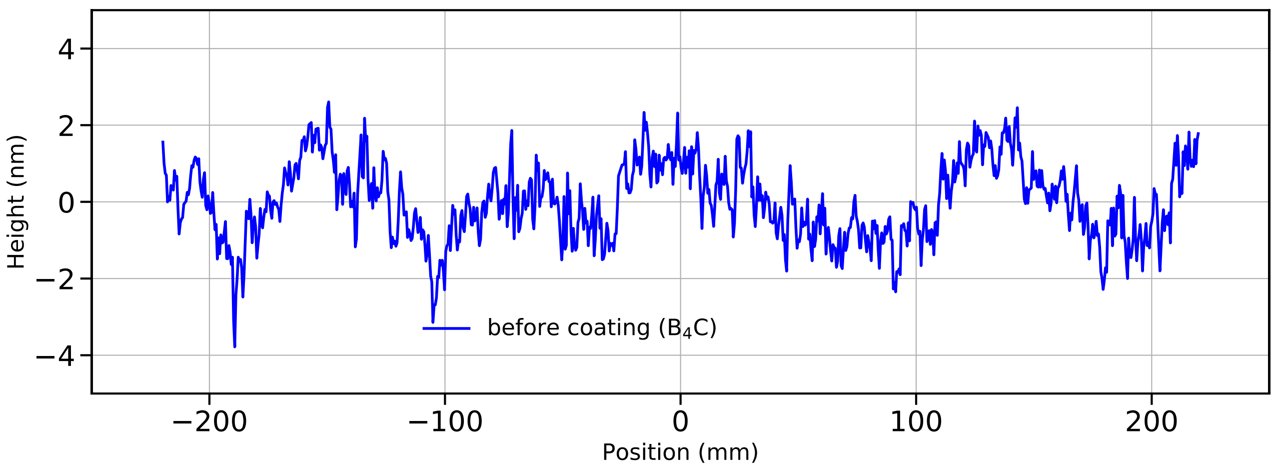

Figure 7. Central profile of the 15-0137 M2 surface before the coating, when 6-power polynomial fitting is removed.

4. MEASUREMENTS AFTER COATING

The mirrors are coated at Helmholtz-Zentrum Geesthacht, Institute of Materials Research. B_4C and Pt coatings are deposited on the mirrors using the 4.5 m-long HZG magnetron sputtering facility, which provides a deposition length up to 1500 mm. The mirror substrates are cleaned under a laminar-flow box and afterwards inserted into the chamber. Two super-polished silicon substrates, with dimensions 50 mm x 20 mm x 10 mm and very low surface roughness of less than 0.1 nm rms (manufactured and measured by Gooch & Housego) are coated together with the longer mirrors. The coating thickness is then measured by X-ray reflectometry using a laboratory source (8048 eV). Typically, the accuracy of the layer thickness determination is about 0.01 nm at a single point. The homogeneity, reproducibility and run-to-run stability of the coating process was determined in previous investigations [4-6]. For the MID mirrors considered in this study the coating thicknesses of B4C and Pt are 102 nm and 100 nm, respectively.

After the coating, the mirrors are measured again with the same instrument and method. We report here the measured profiles, compared to the previous ones and to the FEA profile, for both mirrors (Fig. 8 and Fig. 9).

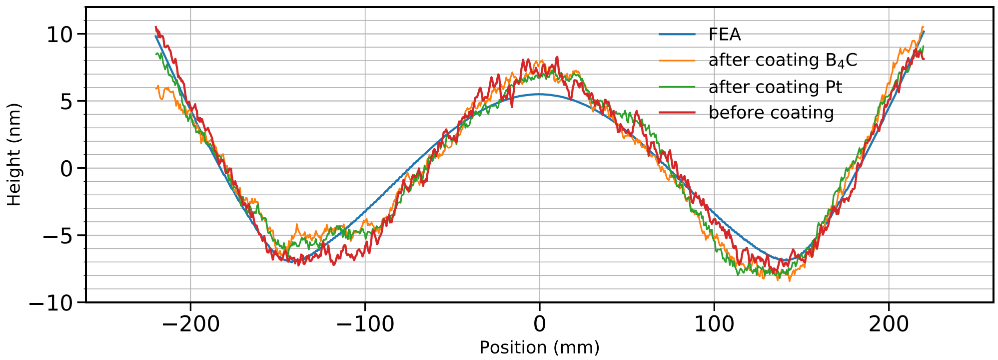

Figure 8. Height central profile measured before and after the coating, for mirror M1, after the removal of the best fitting parabola, compared with Finite-Element-Analysis profile (FEA).

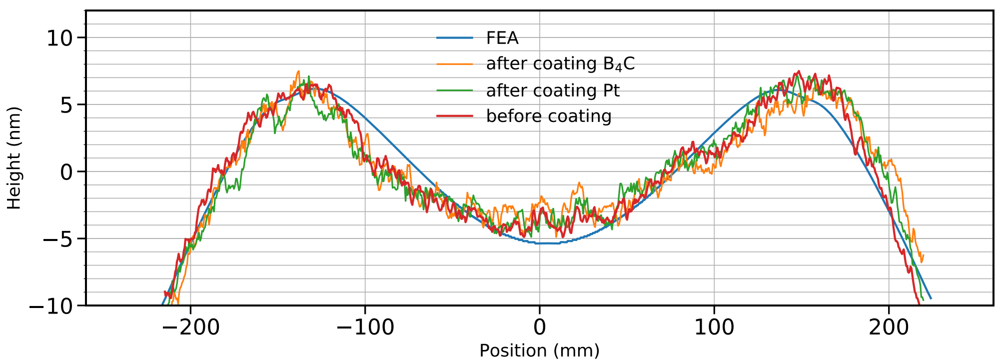

Figure 9. Height central profile measured before and after the coating, for mirror M2, measured in grazing incidence and with the removal of the best fitting parabola, compared with Finite-Element-Analysis profile (FEA).

5. CONCLUSIONS

The present work compares high accuracy profilometric measurements before and after the coating, made to check that the coating keeps the high quality of the mirror polishing. In this aspect, we can clearly see the minor impact of the coating when comparing the profiles reported. The coating process appears to be very much under control and suitable for ultra-high precise mirrors, even for X-Ray Free Electron Laser applications where the specifications are very demanding. Further data analysis of the data is on-going, in particular to verify the macro-shape and the impact of the coating in the different spatial frequencies. The mirrors have been installed recently inside a UHV chamber (designed and manufactured by AXILON AG) situated in the experimental hutch of MID where they will be used for the upcoming experiments.

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